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SYNTHETICAL CARBOLIC ACID.

By H. W. Jayne.

Read at the Pharmaceutical Meeting of the Philadelphia College of Pharmacy, Nov. 17.

It has been known for some years that carbolic acid could be produced by numerous synthetical methods, but interest in the subject has been especially manifested recently on account of several firms, in response to the constant demand for purer preparations, having undertaken its manufacture on a commercial scale. Of the many reactions by which phenol can be produced, only two are, at present, commercially practical; both using pure benzol as the starting point.

The first or sulphonate method is applicable to the preparation of all phenols and has been used for some years, producing on an immense scale naphthol, the phenol of naphthalin. In this method pure benzol, free from thiophene, is placed with about five times its weight of strongest commercial sulphuric acid (67° B.) in closed cast-iron pots, provided with stirrers and lead coolers and capable of being heated by a steam jacket. While the mixture is slowly stirred, the vessel is gently heated with steam in such a manner that the vapors of benzol which pass into the cooler are continually returned to the kettle. After a number of hours the reaction is finished, and the benzol not acted on, is collected as it flows from the cooler. The crude benzol-sulphonic acid, mixed with the excess of sulphuric acid used, is allowed to cool and then diluted with water in a lead-lined tank. Slaked lime is added to the hot solution in sufficient quantity until it is faintly alkaline. This removes the excess of acid by forming calcium sulphate, which is then filtered

off by means of a filter press. The clear liquor containing calcium benzolsulphonate is treated with sufficient sodium carbonate to precipitate all the calcium as carbonate, which is removed by filtration, and the liquor is now evaporated to dryness, leaving the sodium benzolsulphonate as a white powder.

In a large cast-iron kettle, heated by a coal fire, caustic soda is melted, and small portions of the dry sodium salt, prepared as above, are gradually added and finally the whole is kept in quiet fusion for some time. The melt now contains sodium carbolate and sulphite together with the large excess of caustic soda used—

$$C_6H_5 — SO_3Na + 2NaOH = C_6H_5ONa + Na_2SO_3 + H_2O$$

It is ladled from the kettle into pans and allowed to cool, broken up, dissolved in water and acidified with sulphuric or hydrochloric acid. The phenol thus liberated separates from the concentrated salt solution, and can be collected and distilled.

In melting the sodium benzolsulphonate with caustic soda it is necessary, in order to obtain a good yield, to use a very large excess of the latter. A greater yield is obtained with caustic potash, and if as large a quantity as six parts are used to one of the soda or potash salt a nearly theoretical yield can be obtained, but as this would greatly increase the cost, caustic soda is used instead.

It has been proposed to treat the melt after dissolving in water with carbonic acid gas, which would liberate the phenol equally well as a stronger acid, and in addition would form carbonate of soda or potash, which together with the sulphite already present could be converted into the hydrate by treating with lime, concentrated, and used for a second operation. It does not appear, however, that this has been carried out in practice.

The second method is much simpler. A pure aniline oil, preferably that grade called aniline for blue, is dissolved in water in a lead-lined tank covered with a hood and provided with stirrers and leaden steam coils. The solution is acidulated very strongly with sulphuric acid, and to the hot liquid a solution of commercial nitrite of soda is gradually added, phenol being at once formed.

In this reaction the sodium nitrite, in contact with the acid solution, liberates nitrous acid, which forms diazobenzol sulphate with the aniline sulphate—

$$(C_6H_5NH_2)_2H_2SO_4 + 2HNO_2 = (C_6H_5N = N)_2SO_4 + 4H_2O$$

but as the solution is hot it at once decomposes into phenol with evolution of nitrogen—

$$(C_6H_5N = N)_2 SO_4 + 2H_2O = 2C_6H_5OH + H_2SO_4 + 4N$$

Neither of these synthetical methods can, at the present time, compete in price with the extraction of carbolic acid directly from the coal tar oils.

A good grade of crystal acid can be purchased abroad in large quantities at this time at about eleven cents per pound, while the pure benzol used in the first method is worth at the English refineries about fourteen cents per pound and aniline oil about twenty cents, without taking into consideration the other expensive chemicals necessary to carry out the reaction.

When the synthetical acid was first placed upon the market it excited much interest, and purchasers were willing to pay the high price it commanded, believing that they were getting a much purer article than could be produced by the ordinary methods. This interest has, however, considerably abated since the acid has been found to redden just as easily as the best commercial grades.

It could scarcely be expected that an acid obtained by either of the complex reactions just described would not be contaminated by products formed by side reactions in the process. In its preparation by the sulphonate method, sulphur compounds (thiophenols, etc.) are likely to be formed; and its manufacture from a substance like aniline, which so readily produces coloring matters, could scarcely be carried out without at the same time forming bodies which at once, or later under the influence of light and air would discolor it.

In addition, commercially pure benzol or aniline oil always contain small quantities of, respectively, toluol or toluidine. These bodies being submitted to the same treatment as their homologues give cresylic acid. It is true that this acid would be present only in minute quantities, but sufficient to reduce the melting point of the resulting carbolic acid.

Lunge has shown that the addition of 1·3 per cent. of cresylic acid to pure phenol reduces the melting point eight degrees to 32½°, and in the preparation of a high grade carbolic acid a difference of a part of a degree is of great importance.

Forty degrees acid is at present a commercial article sold at excessively low prices, and if a small part of the attention and labor

which is used in producing a synthetical acid was expended in the further purification of this 40° acid, without doubt just as good if not a purer article could be produced direct from tar oils and at a very much lower cost.—Laboratory of the H. W. Jayne Chemical Company, Nov. 14, 1891.

A COLORIMETRIC ESTIMATION OF VANILLIN.

By Frank X. Moerk, Ph.G.

Contribution from the Chemical Laboratory of the Philadelphia College of Pharmacy.—No. 97.

Read at the Pharmaceutical Meeting, Nov. 17, 1891.

"Will natural vanillin give the same color reaction as the synthetical product?" and "Is this test applicable to the assay of vanilla extracts?" were questions asked of me at the last Pharmaceutical Meeting after reading a paper on a new color reaction of vanillin answers to these queries will be given in the following paper.

Without having any doubt that the vanillin of different origin would react similarly, I received from Mr. Beringer a small quantity of vanillin which had been removed from the bean, and with this obtained the same reaction, thus showing by another test the identity of the natural and synthetic vanillin.

To determine the applicability of this test in examining vanilla extracts and their substitutes, Mr. Beringer prepared for me a number of mixtures of vanillin and coumarin, some of which were colored with various vegetable coloring substances. A pure extract of vanilla, largely diluted with water, will give with salts of iron a peculiar violet-brown coloration, which interferes notably with the test, and to remove this disturbing influence it is important to decolorize the extract before proceeding with the development of the characteristic color.

To effect the decolorization various methods were tried. Adding lead nitrate solution to the diluted samples, and then by the addition of magnesium oxide, suspended in water, precipitating lead hydrate, which carried along with it the coloring matter; it was found that by this procedure considerable of the vanillin was also removed from the solution (as much as three-fourths in some cases). Instead of the lead salt, alum and ferric sulphate were then used with somewhat better results, but still with loss of vanillin; agitating the diluted extract with freshly precipitated ferric hydrate was not suc-

cessful because of the filtrate having the peculiar violet-brown coloration; lastly, and with best results, freshly precipitated lead hydrate was agitated with the samples and after standing a short time the mixture filtered.

The lead hydrate is most quickly and conveniently prepared by dissolving one gram lead nitrate in 25–30 cc. water, adding a few drops of phenolphthalein and then a dilute potassium hydrate solution until a permanent pink color results; by the cautious addition of a dilute solution of lead nitrate the pink color is discharged and then the precipitate washed two or three times by decantation, and lastly water added to make 100 cc. of the mixture.

The determinations were made as follows: 2 cc. of the samples were diluted with about 50 cc. water, 10 cc. of the lead hydrate mixture added, and then sufficient water to make 100 cc.; after standing a few minutes the mixture was filtered through a dry filter, 50 cc. removed to a flask of 100 cc. capacity, bromine water added, drop by drop, until after agitation the bromine odor was permanent, then a 1 per cent. ferrous sulphate solution until the intensity of the color was reached, lastly, water added to make 100 cc. and, after standing about an hour, filtered. One cc. extract by this test will form 100 cc. of a more or less colored solution; two standard solutions were made; one containing 0·002 and the other 0·0005 vanillin in 100 cc. of the test; the comparisons were made in large test tubes, the different height of the columns being inversely proportional to the amount of vanillin present. With colorless solutions of vanillin there is no difficulty in accurately determining the amount of vanillin present, but in colored solutions there is a yellowish tinge to the test which renders accurate comparison impossible. While this color reaction is not suited for the accurate estimation of vanillin in extracts and colored substitutes, still it allows a reliable comparison of these preparations; this was found to be the case in the samples prepared for me by Mr. Beringer, while the true amount of vanillin would not be given by the test, still I could invariably say which contained more than another. An important point to be noted in this connection is that while the odor of vanillin is destroyed in this test, that of coumarin is not affected, so that the test will serve to give an idea as to the value of a vanilla preparation and also to detect small quantities of coumarin in presence of vanillin.

Two of the samples examined were from reliable manufacturers,

and in these the vanillin was easily recognizable by the bluish-green color produced in the test; a third specimen was made from a bean that had been kept for at least twenty years, and in this the color produced was also characteristic of vanillin, showing that age does not cause deterioration of the bean; with these three samples the completed tests were odorless. A fourth sample obtained from a grocery had a strong odor of coumarin after the completion of the test, while the color of the test excluded more than minute quantities of vanilla in its preparation.

NOTE ON BISMUTH SALICYLATE.

By Joseph W. England, Ph.G.

Read at the Pharmaceutical Meeting of the Philadelphia College of Pharmacy, Nov. 17.

Since salicylic acid is a monobasic acid, true acid salts of it are impossible. What is meant by "acid" bismuth salicylate in commerce is a mixture containing a very varying amount of free or uncombined salicylic acid. It is in no sense a true, definite compound. The existence of the so-called "acid" salt arose through the imperfections of early manufacture, salicylic acid being so readily precipitated from solutions of its salts by acid solutions. Lately there has been an improvement in purity, but the writer believes that there exists still further room for better quality. A prominent firm of manufacturing chemists has kindly furnished me with the following list of analyses made by themselves of bismuth salicylates. The first one examined was of foreign make, the second of domestic origin; both were made in 1883; the others were all made in 1891.

In making comparison it should be remembered that anhydrous normal bismuth salicylate ($BiOC_7H_5O_3$) should yield 64·46 per cent. of Bi_2O_3 on ignition. The yield of Bi_2O_3 of the samples examined was as follows: No. 1 (Foreign acid), *27·2* per cent.; No. 2 (Domestic, basic), *66·3* per cent.; No. 3 (Acid), *43·4* per cent.; No. 4 (Foreign, basic), *67·4* per cent.; and No. 5 (Domestic, basic), *61·2* per cent.

The wide differences in composition will be noted, as well as the steady improvement towards the normal basic salt. If simply bismuth salicylate is ordered, manufacturers supply the basic compound and not the acid, arguing that the former is the true normal compound, and that if the "acid" compound is the more active one, therapeutically, as is believed, it is far less confusing to supply the basic compound, and let the physician add salicylic acid in any pro-

portions that he may wish, than to supply a product containing a very indefinite quantity of free salicylic acid. A process for preparing bismuth salicylate was described in the August number of the "Journal," page 401.

A NEW TABLET MACHINE.

By J. R. Witzel, M. D.

Read at the Pharmaceutical Meeting of the Philadelphia College of Pharmacy, Nov. 17.

Realizing the great demand for compressed tablets, the writer has endeavored to bring before the profession a tablet-compressing machine that is simple, powerful, compact and, as nearly as possible, perfect in every respect. The accompanying cut shows the appearance of the machine, the motions of which are positive and automatic.

The frame is one solid casting; the hand machine weighs about 125 pounds, occupies a counter space of 10 x 17 inches and stands 9½ inches high; power machines have an additional stand to give clearance for the large fly wheel. The capacity is from 55 to 95 tablets per minute, and is increased in proportion to the number of plungers in operation. The pressure can be regulated to any desired degree, is direct and comes to bear between the cams which impart the desired motions to the sliding blocks. The steel

plungers, if necessary nickel plated, have a projecting shoulder on the lower part and may be of any size or shape, and hopper and mould are stationary. There being absolutely no waste, a saving of material results, and the absence of friction from feed makes the machine run easy.

Ample provision is made for taking up lost motion from wear of moving parts. The dies and moulds can be easily changed from one size to another, and the regulations being few and simple, they are readily understood and easily manipulated. The working parts being inclosed, accumulation of dust is prevented, wearing of the parts is lessened and foreign matters are not rubbed into the mould for compression; hence, the tablets are kept clean and unobjectionable for hypodermic medication.

Tablets are formed by the following motions: When the plungers are below the hopper, they receive the proper amount of material, which is conveyed under cover into the mould; one plunger is held stationary, while the other advances and compresses the material into the shape desired; then both dies recede, thus loosening the tablet in the mould and bringing it to the end of the mould, where it is ejected by the wiper.

TACONY, PHILADELPHIA, November, 1891.

NOTE ON FLUID EXTRACT OF TRITICUM REPENS.

BY JOSEPH W. ENGLAND, PH.G.

Read at the Pharmaceutical Meeting of the Philadelphia College of Pharmacy, Nov. 17.

Some time ago my attention was attracted towards the variability in properties of commercial fluid extracts of couch grass. The differences were so marked that an examination was made of a number. In the six samples examined, four different shades of the normal ruby red color were presented. The odor and taste was the same in all cases save one (No. 2), which gave evidence of containing considerable caramel. The specific gravities ranged from 1·069 as the lowest, to 1·154 as the highest. Assuming that the menstruum used in their making was the officinal one, of one part alcohol and four parts of water; the relative value of the fluid extracts in the amount of solids dissolved, ranged from 92·6 to 100; the latter figure representing the highest specific gravity.

The results in tabular form are as follows:

No.	Made.	Sp. gr.	Relative Value in Extractive.
1, ...	Fall, '84	1·112	96·4
2, ...	April, '90	1·154	100·
3, ...	Aug., '90	1·093	94·7
4, ...	Oct., '90	1·085	94·
5, ...	Sept., '91	1·069	92·6
6, ...	June, '91	1·148	99·4

The chemical character of commercial fluid extracts of couch grass is of interest for the reason that it is now being employed quite frequently in certain renal and bladder troubles, with asserted success. What constituent its medical properties are due to is uncertain. Flückiger and Hanbury[1] state that the constituents of couch grass include no substance to which medicinal powers can be ascribed, and that Müller has found the juice of the rhizome to contain 3 per cent. of sugar and 7 to 8 per cent. of triticin, an inulin-like body, amorphous, odorless, tasteless and very hygroscopic and readily transformed into sugar, if its concentrated solution be kept for a short time at 110° C. Triticin, it should be noted, is readily changed by heat, especially in the presence of dilute acids, and since the drug is rich in acid malates, it is probable that this change often ensues in fluid extracts where heat is employed in concentration and there is thus an increase in the percentage of the sugar normally present in the drug. Ludwig and Müller[2] claim that there are three sugars present in couch grass, fruit sugar, levulose and a dextrose not identical with cane sugar. Later, Plauchud[3] found 3 per cent. of crystallizable sugar, 4 per cent. of uncrystallizable sugar and 14 per cent. of starch. Mannite is not constant in its presence, save only probably as a result of fermentation in the extract. Pectin and resinoids are absent.

The presence of caramel in one of the samples examined, probably arose from overheating in drying, a very moist drug containing a large amount of natural sugars, since the preparation, as well as the others, was obtained from reliable houses. The variations

[1] Pharmacographia, p. 730.

[2] Nat. Disp., 1884, p. 1553.

[3] Zeitschr. Oest. Ap. Ver., 1877, p. 457; vide Proc. A. P. A., 1878, p. 181.

in the percentage of extractive, probably arose to a large extent, from the fact that the pharmacopœial injunctions to use spring collected couch grass was not observed.

MULLEIN OIL.

By George M. Beringer.

Read at the Pharmaceutical Meeting of the Philadelphia College of Pharmacy, Nov. 17.

The object of the present note is to call attention to some of the products in the market under this name. The pharmacopœias and dispensatories of a century ago nearly all contained formulas for *Oleum Verbasci*, the formula given in the Universal Pharmacopœia directing to digest, at a gentle heat, mullein flowers one part, in olive oil two parts, until extracted, and then express.

The National Dispensatory (p. 1601) evidently refers to a similar preparation, stating that olive oil saturated with mullein flowers during prolonged exposure to the sun or fire for several days in a corked bottle is a popular remedy in Germany for bruises and is used for bleeding piles. Its use has recently been revived in this country. The best results are obtained from fresh flowers. In the absence of these, a good preparation can be obtained by using the commercial dried mullein flowers, which are imported from Germany, by the following process:

Take of dried mullein flowers one part; alcohol one part; macerate for a few hours and then add olive oil four parts, digest on a water bath for several hours until the alcohol is entirely evaporated and the extraction is completed, then express.

A prominent homœopathic pharmacy has recently advertised mullein oil as a remedy for earache, deafness, difficult urination, enuresis, coughs, and as an application to inflamed surfaces, ulcers, piles and sore eyes. Their article was advertised in ½ oz. bottles at 60 cents; 1 oz., $1.00. A sample of this "valuable product," in original vial, was procured by the writer. It was a thin liquid of a brown color, with the pleasant odor characteristic of the dried German flowers. It had an acid reaction to test paper and showed a specific gravity of ·9815. The stain on bibulous paper was neither oily nor resinous in character. It was miscible with water, the solution becoming but slightly opalescent. It formed a perfectly clear solution with alcohol and diluted alcohol. It was not miscible with benzin, ether or chloroform. On evaporation on the

water bath three per cent. of residue remained and on incineration 0·3 per cent. of ash was obtained. This preparation was apparently only a weak alcoholic tincture containing a small quantity of acid, most likely acetic.

A sample of another product, prepared by a prominent essential oil house in New York, was also procured for examination. It was a neutral limpid liquid of a bright green color, had a specific gravity of ·835. On evaporation on the water bath 3·724 per cent. residue was obtained and 0·267 per cent. ash was left on incineration. The stain on bibulous paper was resinous. With water an opalescent mixture was produced, which after standing deposited a green precipitate. With strong alcohol it yielded a clear solution without any precipitation, but with dilute alcohol the solution was not clear. With chloroform it yielded a clear solution. Its action with ether precluded it from being ethereal extract or oleoresin, as it formed a clear solution with concentrated ether and a cloudy one with commercial U. S. P. ether and from both of these solutions there was a slight precipitate of a brown color deposited.

The preparation is evidently only a tincture prepared with strong alcohol from the flowers, and from the amount of chlorophyll present, I would suppose that these had been admixed with a portion of the leaf or possibly the calyx.

Neither of these preparations have any claim to the title of an oil under the generally accepted definition of that term, and it is a matter of surprise that such reputable houses should lend themselves to this form of petty deception to introduce an article at exorbitant prices. Another objectionable feature, is that if such preparations are advertised under the same name as á preparation known for a century, an element of doubt is introduced as to the physician's intention in prescribing. As many of our physicians see only the advertisements and do not know the preparations, it is doubtful if they could decide which they really intended.

ON THE PRODUCTION OF OIL OF BIRCH.

By William H. Breisch, Ph.G.

From an Inaugural Essay presented to the Philadelphia College of Pharmacy.

The oil of birch was first distilled in Luzerne County, Pa., about the summer of 1865, shortly after that of oil of wintergreen, and now the manufacture of oil of wintergreen is almost entirely super-

seded by that of the former, owing to the difficulty experienced in procuring pickers of the wintergreen leaf. There are, within an area of six or eight miles, not less than five or six distilleries which produce annually between three and five thousand pounds of the oil. After being distilled, it is either brought to White Haven, the nearest shipping point, and sent to New York parties; or else it is sold to the merchants of the town and sent by them to New York.

The manufacture is quite a simple one.

The wood gathered for the purpose is the black birch saplings; another variety, the white birch, not being used, owing to its very small yield of the oil. The saplings are gathered principally in the summer months, the yield being the greatest at that time; but they are also gathered in the winter. The birch is cut into small pieces, varying from two to four or five inches in length, by a very ingenious device of home manufacture, which consists of one or more knives fastened to the end of the shaft, while the other end is connected with a large wheel, and by means of a belt fastened to a water wheel. The birch is pushed under the knives, by hand, through a box or trough similar to that of a hay cutter, and when cut it is ready for the still.

The still is box-shaped, about eight feet long, four feet wide and four feet high, with a copper bottom, and stayed with bolts. The head of the still is made of copper and connects with a circular worm made of copper or sometimes of tin, placed in a barrel which is filled with water, with a continuous stream of water flowing through it by which the vapors that come over are condensed. The still is filled with the birch wood to within about twelve inches of the top, and a sufficient quantity of water is then added to cover the wood. This is allowed to macerate from eight to twelve hours. The fire is then started and the distillation commences and continues for about eight or sometimes ten hours; but during the first two hours ninety per cent. of the oil has passed over.

For collecting the distillate a common fruit jar is generally used, fitted with a cork having two holes; a small funnel is put into one of the holes, so that the beak of the funnel is about two inches below the bottom of the cork, and connected with the other hole is a suitable pipe forming an outlet.

The distillate passes through the funnel into the receiving vessel,

where the water and oil separate, the oil going to the bottom being heavier; and the water being lighter and in excess, passes through the pipe into a larger receiver, where it is reserved for the maceration of the next quantity of birch to be distilled. This is repeated a number of times before fresh water is used.

The yield, which is about one per cent., is most abundant during the months of July and August.

On taking a sample of the oil from the receiver only a short time after the separation of the water, I found the specific gravity to be 1·17; another sample from the same distillate, which was allowed to stand between 48 and 60 hours, so as to get more thoroughly separated, had the specific gravity 1·18, which I think conclusively proves that the lower specific gravity of pure oil of birch is due to water imperfectly separated.

The color of oil of birch when first distilled is very light or almost colorless, but on standing a while becomes very much darker. Occasionally, the oil is found to be highly colored, due to traces of iron from the oxidation of the tin worm or the can with which the oil comes in contact.

There are three ways of clearing the oil: by decolorization, filtration and redistillation. The easiest method is decolorization, by putting the oil in a bottle, adding a few crystals of citric acid and agitating occasionally, until the oil is colorless, or nearly so.

See also paper on the distillation of oil of birch in Amer. Jour. Pharmacy, 1882, p. 49.

SALIX LUCIDA, *Muhlenberg.*

Shining Willow.

By Robert W. Beck, P.G.

Contribution from the Chemical Laboratory of the Philadelphia College of Pharmacy, No. 98.

This species of willow is a shrub or small tree ten to twenty-five feet in height, and is found along the banks of streams from New England to Pennsylvania and westward. According to Gray it is closely allied to the S. pentandra of Europe. The material for the following investigation was collected by the author in the western part of Pennsylvania.

The following is a summary of the results obtained by extracting the finely powdered bark successively with the different solvents:

	Per Cent.
Petroleum ether,	0·37
Stronger ether,	1·30
Absolute alcohol,	4·39
Water,	4·09
Sodium hydrate (·2 per cent.),	4·04
Hydrochloric acid (1 per cent.),	4·48
Chlorine water,	13·82
Cellulose,	37·60
Ash,	5·40
Moisture,	9·70
Loss and undetermined,	14·81
	100·00

The petroleum ether extract was found to consist of a trace of volatile oil, wax and caoutchouc.

Stronger ether extracted an organic acid resin and sufficient salicin to give a red color with sulphuric acid.

Absolute alcohol removed an aromatic brown resin, tannin and salicin.

The usual plant constituents were obtained from the remaining solvents.

In order to determine whether a satisfactory yield of salicin could be obtained from this bark, a number of assays were made.

(1) A decoction of the sample was treated with lead hydrate, filtered and the filtrate saturated with hydrogen sulphide and the lead sulphide removed by filtration. On evaporation no salicin was obtained.

(2) Thirty grams of the powder were extracted with boiling water, twenty grams of lime stirred in, and the solution filtered. The filtrate on evaporation yielded but few crystals.

(3) A hot infusion of one hundred grams of the powder was treated with lime, filtered and evaporated rapidly to a syrupy consistence. The residue was treated successively with several portions of alcohol, and the mixed alcoholic solutions passed through animal charcoal. On concentrating beautiful crystals of salicin were obtained, amounting to 1·09 per cent.

This process was then employed for further assays, by which 0·30 per cent. of salicin were obtained from the leaves of S. lucida; 0·56

per cent. from the bark of S. alba, and 0·73 per cent. from the bark of S. nigra.

The same specimens were examined quantitatively for tannin, with the following results:

Leaves of S. alba,	6·48 per cent. tannin.
Bark of S. alba,	3·58 " " "
Bark of S. alba,	4·26 " " "
Bark of S. nigra,	3·29 " " "

NOTES ON INSECT POWDER.[1]

By F. A. Thompson, Ph.C.

Having had seven samples of insect powder, representing six eastern wholesale firms and one western one for examination, I thought the result of such analysis might be of some interest to this society, and therefore present to you the practical tests as applied.

Mr. P. MacOwen, F.L.S. (Pharm. Journ. and Tran., Feb., 1890, p. 695), of the "Capetown Botanical Garden," points out that plants yielding Persian insect powder are being cultivated in the colony, and it is hoped may be a source of supply of the genuine, as the flowers and powder are largely adulterated; also that *Pyrethrum cinerariæfolium* likes an open, dry soil, not too clayey, as both the seed and plant are killed by excessive moisture.

Mr. G. M. Beringer (Amer. Jour. Pharm., Jan., 1889, p. 1), reports that in 1889, a number of bales of Hungarian daisies were entered at the New York Custom House as insect powder, and were evidently intended as a sophistication of the Dalmatian insect powder. He states that the similarity in size and appearance of the flowers of the Dalmatian powder would easily deceive the careless or unguarded observer. A microscopical examination of the two varieties as whole flowers rendered the distinction comparatively easy, while in the powdered form the identification of the admixture was impossible. The ash of the Hungarian daisy amounted to 9·3 per cent., that of the Dalmatian 6·5 per cent., while the amount soluble in petroleum ether and ether seemed to be about the same. Alcohol extracted more from the former.

In the examination of the various samples, the color of powder,

[1] Read before the Detroit Chemical Society, November 10, 1891.

amount and constituents of ash, and behavior of the chloroform extract towards reagents, were the essential tests taken up, the practical "insecticide" test, as applied to flies and cockroaches, being omitted for want of time and insects.

The color of the samples coincided with the chemical examination, samples one to five being quite alike in color, while number six was of a deep yellow and number seven less so.

The dealer in buying should insist upon securing a fawn or light yellowish brown colored powder and look with suspicion upon the deep and bright yellow ones.

The ash of a genuine insect powder should not run over eight per cent. Anything above this should be examined carefully for some lead or barium salt. The ash should be almost entirely soluble in hydrochloric acid, but if part is insoluble and the clear fluid becomes green upon boiling, H_2S produces a black precipitate and KI a bright yellow crystalline one, it may be accepted as conclusive proof that lead chromate is present.

Recapitulation of analyses:

Name.	Color.	% Ash.	Constituents of ash.	Chloroform Extract			Adulteration.	Microscopical Examination.
				Color.	and Boracic acid for Turmeric.	and Hydrochloric Acid.		
Dalmatian,	Fawn.	6·5	Normal.	Greenish brown.	Negative.	Slightly green.	None.	No foreign starches.
Dalmatian (uncolored)	Yellowish brown.	6·6	Normal.	Yellowish brown.	Negative.	Greenish.	None.	No foreign starches.
Dalmatian (extra),	Light yellowish brown.	6·9	Normal.	Yellowish green.	Negative.	Slightly green.	None.	No foreign starches.
Dalmatian,	Lightfawn.	6·59	Normal.	Brownish green.	Negative.	Greenish.	None.	No foreign starches.
Dalmatian,	Light yellowish brown.	6·2	Normal.	Greenish.	Negative.	Deep green.	None.	No foreign starches.
True insect powder,	Deep yellow.	12·6	Normal and lead chromate.	Pale yellow.	Negative.	Slightly green.	Chrome yellow about 6 %.	No foreign starches.
Dalmatian,	Dark yellow.	26·8	Normal and lead chromate.	Greenish.	Negative.	Distinctly green.	Chrome yellow about 20 %.	No foreign starches.

PARKE, DAVIS & CO., ANALYTICAL LABORATORY, Nov. 6, 1891.

ANALYSIS OF PUMPKIN SEEDS.

By Wm. E. Miller, Ph.G.

From an Inaugural Essay presented to the Philadelphia College of Pharmacy.

The seeds were deprived of their integuments, and these and the kernels ground separately into a No. 40 powder.

The shells yielded to benzin 2·8 per cent. of extract, from which strong alcohol took up a fixed oil, leaving behind a dark reddish-brown mass. By means of ether the powder now gave 0·9 per cent. of extract, a small portion being soluble in water, yielding a neutral solution; and a resinous substance dissolving in alcohol with a greenish fluorescence, leaving an oily residue. The alcoholic extract from the powder, exhausted as described, amounted to 0·5 per cent., and was a greenish-brown solid of a bland oily taste and liquefying at the boiling point of water. The powdered shells yielded 2·17 per cent. of ash, of which 60 per cent. was soluble in water, and about one-half of the remainder insoluble hydrochloric acid.

The ash of the kernel weighed 4·4 per cent. Petroleum benzin extracted from the powdered kernels 33·6 per cent. of fixed oil, which was obtained of a dark reddish color, and having but little odor, but a rank and somewhat bitter taste. The oil is freely soluble in ether, chloroform and in hot absolute alcohol; but in 95 per cent. alcohol it is almost insoluble. With NaOH a soft brownish soap is obtained.

No indications could be obtained of the presence of a glucoside or alkaloid.

FERRIC PHOSPHATE U. S. P. AND FERRIC PYROPHOSPHATE U. S. P.

By Julius Stieglitz, A.M., Ph.D.

In the September number of the "Pharmaceutische Rundschau," Dr. F. B. Power proposes, in an excellent paper on the official iron salts, to change the official method for the quantitative estimation of the iron in ferric phosphate U. S. P. and ferric pyrophosphate U. S. P.

On having a large number of preparations of these salts to examine from the principal manufacturing houses in the country, I have found the present official method for the qualitative examination to be unsatisfactory in many cases. Therefore, I would suggest that a change also in the qualitative requirements be taken into

consideration. The frequent use of pyrophosphate as even a better preparation than the phosphate and the desirability of having the pharmacopœial requirements perfectly reliable in all cases caused me to examine the subject somewhat extensively.

The present pharmacopœia distinguishes the two preparations by the silver salts of phosphoric and of pyrophosphoric acid, which is satisfactory; but by neglecting first to effect the separation of the respective acid from the citric acid, also present, before making the silver test, its directions prove inadequate to any one examining the salts frequently and carefully. Citric acid gives, under the conditions of the test, also a white precipitate with silver nitrate: At the same time I have never had a sample of pyrophosphate of iron U. S. P. which was entirely free from the corresponding phosphate, and yet the contamination is sometimes so slight that the preparations could not be condemned. Now the consequence of the precipitation, with silver nitrate, of phosphoric acid in the presence of citrates, and on the other hand of pyrophosphoric acid in the presence both of citric acid and varying amounts of phosphoric acid is, that the behavior of both salts is nearly identical. By total precipitation a *very pale* yellow salt is obtained, which one is loth to regard as either phosphate or pyrophosphate. By fractional precipitation both preparations give white, yellowish and yellow tints, the phosphate often giving first a white precipitate of silver citrate, and only with a larger amount of silver nitrate a yellow precipitate. The presence of acetic acid makes the reaction yet worse. Phosphate of silver appears to be more soluble in this acid than silver citrate or silver pyrophosphate: If after complete precipitation a little ammonia is added, bright yellow silver phosphate is thrown down both in the case of phosphate and generally of commercial pyrophosphate.

My time was too limited to investigate whether the appearance of phosphate in the pyrophosphate of iron is due to any converting action during the course of preparation; it probably is due to the use of an impure sodium pyrophosphate. But certainly it would hardly be advisable to condemn every pyrophosphate of iron containing a slight amount of phosphate.

In order to do away with this uncertainty, and especially to have a positive reaction for pyrophosphate without the possibility of mistaking the citric acid present for it, I tried several different methods of proceeding.

As for other phosphates, solution of ammonium molybdate in nitric acid forms an excellent reagent for quickly demonstrating the presence of phosphoric acid in such a scale salt. The amount of citrate in the samples tested did not interfere with the reaction. The yellow precipitate must appear in a few moments if phosphate is present. By taking the same amount of the solutions of definite strength, when applying the test, a practised eye can see by the quantity of precipitate after a few minutes whether a true phosphate is present or phosphoric acid only as a slight contamination of a pyrophosphate. In the few minutes the test requires a solution of pure pyrophosphate of sodium acidified with nitric acid gave no precipitate with the molybdanic solution. This test of the salts under consideration is shorter than the U. S. P. test and is better because citric acid does not give any reaction with the reagent used. But it suffers from the same defect inasmuch as the presence of pyrophosphate is also here demonstrated only by the negative result, viz: The absence of any considerable quantity of phosphates.

By careful addition of acid to the aqueous solution of these scale salts, real iron phosphate and pyrophosphate are precipitated. This reaction could serve to remove the citric acid before making the silver test. It was found, however, that the precipitates are very difficult to filter and wash. Even after thorough washing, an examination showed that they still held back citrates, ether chemically or mechanically. So this reaction proved unavailable.

The separation from the citric acid by means of a calcium salt was tried briefly, but proved too unpromising for me to proceed further in this direction.

The test involving the use of excess of magnesium sulphate for separating the acids proved satisfactory; I obtained good results by adding to the alkaline solution of the acids, called "A" below, as obtained from the scale salts, an excess of ammonium chloride, and pouring the fluid into quite a large excess of concentrated magnesium sulphate solution (33 per cent.) The magnesium ammonium phosphate, precipitated in the cold, was identified further, as below, by being converted into silver phosphate. In case iron pyrophosphate was present, even with some iron phosphate, the filtrate from the precipitate of $Mg(NH_4)PO_4$ yielded magnesium pyrophosphate by boiling. The latter was identified by its silver

reaction and the reaction with acetic acid on boiling. This method of proceeding leaves the chemist very much more satisfied than the U. S. P. process, as the former gives us a positive proof for pyrophosphoric acid, and I can recommend it to the attention of the Committee for the Revision of the U. S. P.

I found, however, that a yet more convenient and satisfactory test could be based on the separation of the acids by means of acetic acid, as described in the next article. The test is not affected by the citrates present. A solution of alkali citrate acidified with acetic acid in any proportion gives no precipitate with magnesium sulphate, in the cold or on boiling. At the same time the presence of citrates was shown neither to prevent the precipitation of magnesium pyrophosphate in acetic acid solution by boiling nor to cause the precipitation of magnesium phosphate under these conditions.

The manner of testing the scale salts in question by this method is as follows:

5 cc. of a 20 per cent. aqueous solution of the salt are added to about 9 cc. of a ten per cent. solution of potassa, previously heated to boiling, and the boiling continued for a moment; after cooling and filtering, one can use the filtrate which I will call "A" for the following tests:

(1) If phosphate of iron is before us, to a few cc. of the alkaline filtrate, some ammonium chloride and 1 to 2 cc. of 10 per cent. solution of magnesium sulphate are added. The easily recognized crystalline precipitate of magnesium ammonium phosphate thus produced is best filtered, washed with water and converted into the silver salt. It may be dissolved in a few drops of nitric acid, ammonia added just till the precipitate reappears and the precipitate taken up with a drop of acetic acid. Addition of silver nitrate solution now produces, if the salt examined is really iron phosphate, a bright yellow precipitate of pure silver phosphate, not mixed with white silver citrate. A practised chemist will not have to convert the magnesium precipitate into the silver salt, easily distinguishing the crystalline $Mg(NH_4)PO_4$ from the amorphous pyrophosphate which would be thrown down if the original salt were pyrophosphate of iron. If the quantity and nature of the precipitate of $Mg(NH_4)PO_4$ or a whitish color of its silver salt makes the chemist suspect that by an error pyrophosphate of iron has been furnished, he can test another portion of the alkaline filtrate "A" for pyrophosphates in the manner just to be described.

(2). If the salt to be tested purports to be pyrophosphate of iron U. S. P. a portion of the alkaline filtrate "A" is tested first for contaminating phosphates, by the method described above, only using instead of an excess of solution of magnesium sulphate, two to four drops of the same. In this way magnesium pyrophosphate, which is precipitated at the first admixture of the reagent, redissolves in the excess of alkali pyrophosphate, while phosphates, if present, are precipitated as magnesium ammonium phosphate. A good preparation should not yield more than a very slight turbidity of phosphate. If a precipitate is formed, it is filtered after five minutes, washed and converted into the silver salt. In this way the admixture of one part of phosphate to nine parts of pyrophosphate of iron could be easily detected.

(3). Another part (3 cc.) of the alkaline filtrate "A" is now tested for pyrophosphates by neutralizing with acid and then adding an excess of five to six drops of acetic acid (36 per cent.) and 1·5 cc. magnesium sulphate solution. On boiling the clear solution, magnesium pyrophosphate is precipitated, if present, while if the salt was a phosphate no precipitation occurs. For further identification the precipitate can be filtered hot and washed with hot water till the washings are neutral. If it be dissolved in a drop or two of nitric acid, reprecipitated with ammonia and redissolved with a drop of acetic acid, silver nitrate will produce white earthy pyrophosphate of silver. A very slight yellowish tinge may occur from insufficient washing, but there is no possible mistaking of the precipitate, the citric acid having been removed from the solution. For further particulars of this test reference may be made to the next article. In this manner the presence of pyrophosphate is proved beyond doubt, the demonstration resting on positive reactions. The whole examination can be completed very nearly as quickly as the present U. S. P. test. Samples have been tested showing contaminations with phosphates which we have had no hesitation in accepting, having proof positive of the pyrophosphate present. The samples have also been examined which were sold as pyrophosphate U. S. P., but which proved to be phosphate, by the U. S. P. test, the white precipitate of silver citrate seemed to be proof of pyrophosphate. The yellowish and yellow tints also obtained by fractional precipitation were no unusual accompaniments of the pyrophosphate of silver; but by the test described no pre-

cipitate could be obtained of pyrophosphate of magnesium. The quantity of magnesium ammonium phosphate precipitated, and the color of the silver phosphate obtained from it, confirmed the result of the pyrophosphate test.

I would therefore suggest that either the method of separating the acids by means of the acetic acid reaction, or of the excess of magnesium sulphate solution, be adopted for the U. S. P. The acetic acid test appears to me to be preferable and the handier to work.

DETROIT, ANAL. LABORATORY OF PARKE, DAVIS & CO.

NOTES ON PYROPHOSPHORIC AND PHOSPHORIC ACID.

BY JULIUS STIEGLITZ, A.M., PH.D.

The general text books of pharmaceutical laboratory libraries give very little information concerning methods for distinguishing pyrophosphoric and phosphoric acid in presence of each other, although their special reactions are generally briefly noted. Such short notices are of little help in the course of work, for special phenomena, limits, and favorable and unfavorable circumstances attending reactions are often as important as reactions themselves. In seeking a practical reaction for quickly and easily separating the two acids for the purposes mentioned in the previous article, I found the usual reactions in very different degrees serviceable.

The test most commonly employed is the silver test. Of course, without separating the acids, a small percentage of pyrophosphate in phosphate does not perceptibly change the color of the silver precipitate, and can therefore not be detected by it. A larger percentage (50 per cent.) very distinctly changes the usually bright yellow precipitate of silver phosphate to a very pale yellow or a yellowish white. On the other hand a very small percentage (1 to 5 per cent.) of phosphoric in pyrophosphoric acid can be detected by precipitating in acetic acid solution with an excess of silver nitrate, filtering from the white precipitate and adding to the filtrate a drop of ammonia. Bright yellow silver phosphate will then appear, undimmed by the pyrophosphate.

The test with molybdanic acid in nitric acid solution is very convenient for finding a small percentage of phosphate in pyrophosphates. The result of the test must be taken at the end of a few minutes, as

pure pyrophosphoric acid is slowly converted even in the cold by the nitric acid employed in the test.

The solubility of magnesium pyrophosphate in alkali pyrophosphate gives a convenient test for demonstrating pyrophosphate in the presence of only a small proportion (Ca. 10 per cent.) of phosphate, even in the presence also of an acid like citric acid which has a white silver salt. If to an ammoniacal solution of the acids named, some ammonium chloride and only a few drops of magnesium sulphate solution are cautiously added as long as the original precipitate of magnesium pyrophosphate redissolves, magnesium ammonium phosphate will slowly separate out, especially on rubbing with a glass rod. On filtering after a little while and boiling the filtrate, a heavy precipitate would indicate pyrophosphate, which can now be tested with silver nitrate without the disturbing influence of either of the other acids. This process is naturally very limited, presupposing, as was said, only a very small percentage of phosphates. If considerable phosphate is present, magnesium pyrophosphate would be precipitated together with the phosphate by the addition of sufficient magnesium sulphate, the remaining alkali phosphate being insufficient to keep the magnesium salt in solution. This method is best combined with the previous method for testing and identifying pyrophosphates.

The fact that magnesium pyrophosphate dissolves in excess of magnesium sulphate and is not precipitated from such a solution by ammonium salts, forms a foundation for another well-known method for separating the two acids. The method was found practical, but a very large excess of magnesium sulphate was required for dissolving the pyrophosphate of magnesium. On adding concentrated magnesium sulphate solution (20 G salt in 60 cc. H_2O) to a solution of sodium pyrophosphate, one volume of the former solution effecting total precipitation of the pyrophosphoric acid, 15 volumes were required to redissolve the precipitate. On the other hand, by pouring the solution of sodium pyrophosphate into concentrated magnesium sulphate solution, an excess of eight volumes of the latter was required to give a clear solution. This very large excess of concentrated solution of magnesium sulphate makes the test rather inconvenient; but it is reliable and otherwise practical.

The quickest and most convenient method for separating the acids for qualitative work appears to me to rest in their behavior

towards acetic acid. The pyrophosphates of the alkaline earths are stated to be difficultly soluble in acetic acid: magnesium pyrophosphate, the salt I worked with, proved comparatively easily soluble in the acid when freshly precipitated in the cold, although much less easily than the phosphate salt; its precipitation is prevented with still less (about half the quantity) acetic acid if the latter is added before the addition of magnesium sulphate. But I found that on boiling such an acetic acid solution of magnesium pyrophosphate, the latter is easily precipitated, unless strong acid is used in very large excess. Magnesium phosphate is precipitated by boiling from solutions containing only the *slightest* excess of acetic acid, but is not precipitated at all from solutions properly acidified. The pyrophosphate precipitated by boiling from acetic acid solution is very much more difficultly soluble in this acid than when precipitated by any means in the cold, and then may even more properly be called "difficultly soluble." But the phosphate obtained by boiling in the presence of very little acetic acid is exceedingly easily soluble in the latter, even in the cold. The method is applied by adding to the solution of the alkali salts an excess of acetic acid (1 to 10 mg. for every 1 mg. P_2O_5 present) and boiling. Phosphate of magnesium will remain dissolved and the pyrophosphate be precipitated. The latter is washed with hot water and can then be used in the pure state for any further test desired. To convert it into the silver salt, it is, on account of its difficult solubility in acetic acid, first treated with nitric acid and ammonia and then easily dissolved in acetic acid before the addition of silver nitrate.

In a solution containing one part of P_2O_5 as $H_4P_2O_7$ to 20 parts of P_2O_5 as H_3PO_4, the pyrophosphate could be easily detected by this method without special precautions. From a solution containing only one part (50 mg) of P_2O_5 as $H_4P_2O_7$ to one hundred parts of P_2O_5 as H_3PO_4, the pyrophosphate was separated pure by this process after making two fractional precipitations by using an insufficient quantity of acetic acid for keeping all the phosphate in solution on boiling (for the first precipitation only 1 mg $C_2H_4O_2$ for 4 mg. P_2O_5 present was used). A sufficient quantity of pyrophosphate was obtained for further tests. This compares very favorably with my experience with the preceding method.

The precipitation of magnesium pyrophosphate by boiling its

acetic acid solution (see above) serves also as a convenient qualitative test for distinguishing the phosphate and pyrophosphate after separation.

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PREPARATION OF PULQUE IN MEXICO.

By ALFONSO MENDIZABAL.

The agave plantations in Mexico are laid out in the following manner: Agaves of a height of about 1 meter are bought and left lying on the field or elsewhere for six months. Then holes of about 2 square meters are dug in the ground, the soil therein is manured, and the agaves are planted. It requires 7 to 8 years for them to fully develop, and during this time they must be manured twice yearly.

As soon as the agaves have reached the above age, they can be used for the preparation of the Mexicans' national drink, "pulque." The manipulation is in this manner: first the middle of the plants, that is the point of vegetation, with the younger portions are cut away. If these, however, are in too advanced a state, it is not possible to prepare the "pulque" from them. After the agave has been cut in this manner, it is not touched for eight days, but after that time, the place where the excision was made, is scratched and scraped, until a sweet aqueous juice, called "aguamiel," or honey-water, begins to flow; the scraping is done twice daily, in the morning about eight o'clock, and in the evening about six.

This water must be collected in containers of ox hide, as it spoils in contact with wood or metal. The juice is now left in the closed containers for eight days, by which time "semilla," probably by a process of fermentation, is produced; then one-half of this fermented juice, contained in the vessel is replaced by fresh aguamiel, and the beverage "pulque" is completed. It never keeps longer than two days, and must therefore be used at once; the number of vessels is always doubled, one half is sold, the other half retained, and is afterward brought back to the former volume by the addition of fresh aguamiel.

In this work it is very necessary that all utensils used be perfectly clean; the utensils necessary for the entire process are the following: For the extraction of the aguamiel a sort of a pipette or suction pipe called "acocote" is the instrument used; this is

apparently the fruit of a cucurbitacea, a tube which bulges out at the lower end, into which a hole is pierced. The aguamiel is extracted by sucking at this hole after the opposite pointed end has been inserted into the incision; when the vessel is full the little opening is closed with the finger, and the contents poured into the leather bags.

For the scraping of the agaves, a rasp (raspador) of the shape of a spoon is used. The vessels of hide in which the beverage is transported from the field to the habitations are called "bolambres." —Translated from *Gartenflora*, October 1, 1891, p. 525, M. A. M.

ABSTRACTS FROM THE FRENCH JOURNALS.

TRANSLATED FOR THE AMERICAN JOURNAL OF PHARMACY.

ANTISEPTIC AND SEDATIVE TREATMENT OF HEMORRHOIDS.—This treatment has been successfully used by Dr. Kassobudski for external and also internal hemorrhoids. (1) For internal hemorrhoids: chrysarobin 1 gm., iodoform 30 cg., extr. belladonna 50 cg., cacao butter 20 gm.; to be made into six suppositories. One suppository to be used a day. (2) For external hemorrhoids: a lotion of corrosive sublimate (1 : 1,000) or phenol (1 : 50) is used several times a day, succeeding which chrysarobin salve is applied, made of the same ingredients as the above suppositories, except that cacao butter is replaced by vaselin 30 gm.—Revue d. Thérapeut., 1891, p. 49.

COMBRETUM RAIMBAULTII, *Heckel.*—This arborescent plant is indigenous to Western Africa along the coast, where it is known as *kinkeliba*, the leaves being used in hematuric biliary fever, to which the whites and also the natives of those climates are subject. The plant is furthermore used in cases of severe colic and to arrest vomiting, and the fruit in treating purulent ulcers. The leaves, in the form of decoction (4 : 250), are said to be strongly tonic, diuretic, to produce emesis at first, but then to prevent the return of the same, and lastly to call forth a biliary diarrhœa.

Petroleum ether extracts 2·66 per cent. of a yellow waxy matter; alcoholic extraction yields 27·12 per cent. of substances consisting principally of tannin; inorganic constituents (ash) present about 3 per cent.—Heckel and Schlagdenhauffen, *Nouveaux Remèdes*, 1891, p. 229.

SULPHATE OF SPARTEINE AND IODIDE OF SODIUM.—In endeavoring to make a solution of 2 gm. of the latter and 20 centigrams of the former in 120 cc. of water, L. Julliard (*Union Pharmaceut*) found that if the salts are mixed and then the water added, a clear mixture at first results, which, however, is quickly blackened by the formation of a precipitate, which takes some time to settle. If solutions of each are made and then mixed, the liquid remains clear only a short time, gradually becomes cloudy and of a dirty yellow, and after a few hours a black colored precipitate collects on the bottom of the vessel. This dark precipitate is not iodine, but very probably iodosulphate of sparteine. The iodide used was alkaline, while the alkaloidal salt was acid in reaction.

LINAMARIN.—A. Jorissen and E. Hairs (*Acad. roy. de Belgique* (3) **21** (1891), 529) have isolated a glucoside, linamarin, from the germs of *Linum usitatissimum*. The germs, coarsely powdered, were treated repeatedly with boiling 94 per cent. alcohol, the latter recovered and the residue taken up with warm water. The resin and fat are separated and the aqueous solution treated with a slight excess of lead acetate. After filtration and precipitating the lead with H_2S the liquid is evaporated to a syrupy consistency. This residue is extracted with boiling alcohol, the solvent recovered for the greatest part, and the remaining liquid mixed with ten times its volume of ether under constant agitation. The residue remaining on distilling off the ether is taken up with water and this solution concentrated. Standing over sulphuric acid for some time the concentrated solution is converted into a crystalline mass of linamarin. For purification it is again treated with ether and alcohol as above. Lastly, the principle is dissolved in two parts of warm absolute alcohol, and the solution cooled under agitation. The germs yield about 1·5 per cent. of the glucoside, which forms colorless needles possessing a refreshing but very bitter taste, is soluble in water and alcohol, but almost insoluble in ether. Concentrated sulphuric acid does not color it; dilute mineral acids decompose the glucoside into hydrocyanic acid, a fermentable sugar reducing Fehling's test, and a volatile compound possessing some characters of ketones, and giving with iodine and potassium hydrate the iodoform reaction. Boiling barium hydrate liberates ammonia. Linamarin contains C 47·88 per cent., H 6·68 per cent., N 5·55 per cent., O 39·89 per cent.

CRYSTALLINE PRODUCTS OF LEMON AND BERGAMOT OIL.—L. Crismer (*Bull. Soc. chim.* (3) **6** (1891), 30) treated the residue from the distillation of oil of lemon at 10 mm. pressure with petroleum ether, until a precipitate began to form and put the mixture aside in a cool place. After a few days warty crystals had formed which were recrystallized from absolute ether. The crystals fuse at 143–144° C. (289–291° F.), are colorless and odorless. Concentrated sulphuric acid colors it yellow; on addition of a drop of nitric acid a green color, with a trace of permanganate of potash a blue changing to a green is developed. The formula of these crystals is $C_{10}H_{10}O_4$ (isomeric with Hoffmann's hesperetinic acid). The petroleum ether used above leaves a butyraceous mass on evaporation which on purification with alcohol melts at 50° C. (122° F.). Oil of bergamot treated in a like manner yields a white substance fusing at 183–184° C. (371–373° F.).

CHANGE IN MOULDED NITRATE OF SILVER IN CONTACT WITH SEEDS.—A. Barillé (*Rép. de Pharm.* (1891, **47**, 403), has examined the cause of the change in moulded silver nitrate when kept with coriander or linseed to prevent breakage. The change which takes place consists in the corrosion of the sticks and the blackening of the seeds. The cause seems to be the presence of volatile and fatty oils or mucilaginous compounds, these causing decomposition in the silver salt especially in light. The author recommends the use of powdered pumice stone for preservation.

PHYLLANTHIN.—M. Ottow (*Nederl. Tijds. voor Pharm.*, 1891, **3**, 128), isolated from *Phyllanthus Niuri*, euphorbiaceæ, indigenous to Java and there used in medicine, a bitter principle *phyllanthin*, $C_{30}H_{37}O_8$. This crystallizes in colorless needles or flakes, possesses an intensely bitter taste and is almost insoluble in water, easily soluble in alcohol, petroleum ether, ether, chloroform, benzene and glacial acetic acid. At 200° C. (392° F.) it is volatilized and condensed as an amorphous mass in the cooler portions of the vessel. In a few days the amorphous variety changes to the crystalline state.

EXPLOSIVE COMPOUND OF BARIUM AND CHROMIUM.—E. Péchard (*Compt. rend.*, 1891, **113**, 39), obtained an explosive body on mixing hydrogen peroxide in excess with a cold, aqueous solution of chromic acid (8 gm. to 1 L.) and adding to this a cooled solution of baryta until an alkaline reaction is obtained. A precipitate is formed which is yellowish, but gradually changes to brownish. As

soon as an evolution of oxygen begins the mixture is thrown into a large amount of water. The precipitate is washed by decantation, and then dried over sulphuric acid. On heating this body detonation takes place, a mixture of barium chromate and baryta remaining behind; water has no action on the body; sulphuric acid causes a rapidly disappearing blue color. The composition of the body is possibly $BaCrO_5 + \frac{1}{10} BaO_2$.

Oil of lemon.—V. Olivieri (*Gazz. chim.*, xvi, 318) found in oil of lemon besides the limonene (Wallach), also another terpene $C_{10}H_{16}$ boiling at 170–170·5° C. (338–339°F.), the tetrabromide of which fuses at 31°C. (88°F.), but the dihydro-chloride showing the characteristics of limonene. From the higher boiling portions the author has furthermore isolated a sesquiterpene $C_{15}H_{24}$ boiling at 240–242°C. (464–468°F.), which increases in quantity with the age of the oil. For detecting adulteration with turpentine the author recommends the use of the polarimeter. Lemon oil is lævogyre $(a)_D = -55°$ while oils of turpentine are more or less dextrogyre. (French oil of turpentine is lævogyre).

Volemite.—M. Bourquelot (Rép. de Pharm., 1891, 338) isolated from *Lactarius volemus*, a fungus, a saccharine body to which he has given the name of volemite. He extracted the fungus with 90 per cent. alcohol recovered the alcohol, and treated the residue with 95 per cent. alcohol. Volemite crystallizes in colorless needles which are radially arranged, fuses at 140°C. (284°F.) and is more soluble in water and alcohol than mannite. It possesses no reducing power, does not ferment, is not altered by dilute sulphuric acid, and does not yield an osazone. Its rotatory power is $a_D = +2·4°$.

Helixin.—G. Joulin (*Jour. de Pharm. et de Chim.*, 1891, **11**, p. 20), has isolated the glucoside helixin from the ordinary ivy, *Hedera Helix*. The leaves and stems of the ivy in the form of a very coarse powder are boiled with water, the decoction filtered and then treated with lead acetate. The precipitate is collected on a filter, washed with cold distilled water and decomposed with sulphuretted hydrogen. The resulting liquid is filtered and evaporated on a water bath to the consistency of an extract and this taken up with 85 per cent. alcohol. The alcoholic solution on spontaneous evaporation leaves helixin, a reddish yellow syrupy mass, possessing an astringent slightly bitter taste. It is acid to test paper, without action

on Fehling's test except on inversion with an acid. Helixin is soluble in water and alcohol, insoluble in ether, chloroform and benzene. The color reactions are as follows: concentrated sulphuric acid, a brilliant red; concentrated hydrochloric acid, yellow; ammonia, yellow; pyridine, olive green precipitate; iron salts, green; sulphocyanide of potassium, rose disappearing quickly; bichromate of potassium and sulphuric acid, red then green; caustic soda, green. The aqueous solution of the glucoside foams just like solutions of saponin.

MIXTURE FOR ASTHMA.—Huchard (*Rev. gén. de Clin. et de Thérap.*, 1891, No. 26) prescribes the following mixture in cases of asthma: Iodide of potassium, Tinc. lobeliæ, Tinct. polygalæ, (*senega*) āā 10 gm. Extr. opium 0·1, Distilled water 900 gm. Dose, one teaspoonful morning and night with ¼ glass of water.

GLEANINGS FROM THE GERMAN JOURNALS.

BY FRANK X. MOERK, PH.G.

Water of ammonia will absorb carbon dioxide from the air, but in doing so there is very little carbonate of ammonium formed, the greater portion going to form carbamate of ammonium. J. Hertkorn, in calling attention to this, states the requirement that water of ammonia should not become immediately turbid when mixed with lime water, is not sufficiently exacting as indicating absorption or presence of carbon dioxide; the test should require that the mixture of water of ammonia and lime water should not show more than a faint turbidity *after boiling*, this procedure changing the carbamate into carbonate. Attention is also called to this presence of carbamate as a source of error in quantitative analysis; if carbon dioxide be absorbed in such a water of ammonia and then precipitated at a boiling temperature as calcium or barium carbonate by the addition of a barium or calcium salt, the results will of course be too high.—*Chemiker Ztg.*, 1891, 1493.

Examination of spices.—At the recent meeting of food-chemists and microscopists in Vienna, the following maximum and minimum figures for the examination of spices were proposed: *Allspice*, not more than six per cent. ash, of which not more than 0·5 per cent. should be insoluble in hydrochloric acid; *cinnamon* should not yield more than 5 per cent. ash, not more than 1 per cent. insoluble in hydrochloric acid; should yield not less than 1 per cent. volatile oil; *cloves* not more than 7 per cent. ash, not more than 1 per cent.

ash insoluble in hydrochloric acid, not less than 10 per cent. volatile oil; *black pepper*, not more than 6·5 per cent. ash, not more than 2 per cent. ash insoluble in hydrochloric acid, not more than 15 per cent. nor less than 12·5 per cent. moisture; *white pepper*, maximum ash percentage 3·5 per cent., 1 per cent. ash insoluble in hydrochloric acid, moisture as above; *saffron*, not more than 8 per cent. ash, 0·5 per cent. ash insoluble in hydrochloric acid, 13–14·7 per cent. moisture, 6–7 per cent. chloroform extract.—*Chemiker Ztg.*, 1891, 1543.

Boric acid, according to Jaenicke, has only a slight disinfecting or sterilizing action, but on the other hand has the power of preventing the growth or multiplication of bacteria etc.; to realize this property of boric acid the wound should be kept filled with a saturated solution of boric acid until it has healed. Better than the boric acid, because of greater solubility, is a compound made by boiling together equal portions of boric acid, borax and water; upon cooling, the compound crystallizes in large crusts which at ordinary temperature will dissolve to form a 16 per cent. solution (boric acid 4 per cent.) or at blood temperature a 30 per cent. solution (boric acid, 7 per cent.); the solution has a neutral reaction, and is more certain and longer in its effectiveness than is boric acid in solution. Because of its non-irritating action, its non-poisonous nature and its unchangeable effectiveness it is superior as an antiseptic to all other substances.—(*Therap. Monatsh.*) *Chem. Repert.* 1891, 289.

Pental is the name under which *amylene* or *trimethylethylene* C_5H_{10} is re-introduced by Prof. v. Mering as an anæsthetic. It is made chemically pure by Kahlbaum, Berlin, from amylene hydrate by heating with some acid; it forms a very volatile, colorless liquid, boiling at 38° C.; it is insoluble in water, but miscible in all proportions with alcohol, chloroform and ether. It is stated to be superior to chloroform and ethyl bromide in that there is no danger attending its inhalation, it is more reliable and there are no bad after-effects.—Holländer (*Therap. Monatsh.*) *Chem. Repert.*, 1891, 289.

Tuberculocidinum is the name given by Prof. Klebs, Zurich, to a purified *tuberculin;* the impurities to be removed are *organic bases* or *alkaloids* which are the cause of the intense febrile reaction. The method followed in its preparation is precipitating the tuberculin with

alcohol, dissolving in water, and extracting the alkaloids by agitation with chloroform (Pictet) or benzol (crystallized); a more recent process depends upon the precipitation of the alkaloids (no reagent or precipitant is mentioned. F. X. M.), and extracting the tuberculocidinum from this precipitate with water; by this process its properties, such as being precipitated by absolute alcohol and ammonium sulphate, also its physiological action, and its behavior towards albuminoidal reagents, are not impaired. The use of this substance is harmless; it does not produce fever, and speedily shows an improvement in the condition of the patients; the hectic fever and night-sweats disappear, the appetite increases, the catarrhal process in the lungs with its symptoms, cough and expectoration, is noticeably arrested, the bacilli in the expectoration become granular and the portions capable of absorbing dyes become smaller and smaller, and finally disappear entirely. In some thirty patients treated no objectionable symptoms could be discovered; to thoroughly test its action, a number of physicians have been supplied with the remedy.—*Deutsch. Med. Wochenschr.; Pharm. Ztg.*, 1891, 700.

Eserine salicylate is readily made from the sulphate by the following process: 100 parts eserine sulphate are dissolved in a suitable quantity of water, and an excess of a solution of sodium bicarbonate added and agitated with several portions of absolute ether; the ethereal solutions are filtered into a beaker containing 35·5 parts salicylic acid dissolved in ether, thoroughly mixed and the eserine salicylate collected upon a filter, washed with absolute ether and dried, protected from sun-light and air. Any excess of salicylic acid that may have been present, is removed by the washing with ether; the theoretical quantity of the salicylate is 106·5 parts; in practice this is a little too high. The success of this method depends upon rapid manipulation and preventing exposure to sun-light, otherwise the salt obtained will be of a red color due to the decomposition of eserine and formation of *rubeserine*; washing the crystals with alcohol will remove any red color but with loss of eserine salicylate. P. Birkenwald, *Pharm. Ztschr. f. Russl., 1891, 657.*

Phenerythen, the red coloring substance of carbolic acid, has been more thoroughly examined by its discoverer, E. Fabini; it forms, when pure, an amorphous, resinous, odorless and tasteless, dull black powder, having the composition $C_{30}H_{30}NO$ and melting at 98 °C.;

it is soluble in carbolic acid with beautiful red color, in ether with yellow, in toluol, ethyl-alcohol, amyl-alcohol and acetic acid with browish-red color; it is difficultly soluble in benzin and carbon disulphite and insoluble in water. Plant and animal-fibres are dyed directly with a brownish-red color. Its formation is explained as follows: If metals act upon carbolic acid containing ammonia there is formed by reduction a quinone-like body of a dirty-brown color, which later by oxidation is converted into phenerythen. Acting like a weak base it forms a sulphate of indigo-blue color, a nitrate of red color and a hydrochlorate of reddish violet color. Phenerythen in an alcoholic, acetic acid solution is decolorized by treatment with zinc dust forming a leuco-base; red carbolic acid in like manner can be obtained colorless by the action of nascent hydrogen, but exposure to air will cause the red color to speedily reappear. —(*Pharm. Post*, 1891, 903) *Pharm. Ztg.*, 1891, 701.

Gallacetophenone, a substitute for pyrogallol in dermatology, is recommended by v. Rekowsky; it is also a triatomic phenol having the formula $C_6H_2(CH_3CO)(OH)_3$ (pyrogallol $C_6H_3(OH)_3$). In alkaline solutions it is, in distinction from pyrogallol, only slowly oxidized; it is relatively non-poisonous. Only slightly soluble in cold water (0·18 parts in 100 parts), it is easily soluble in boiling water, alcohol and ether; in glycerin it is soluble in all proportions. The solubility in cold water can be increased to 4 parts in 100 parts if it be dissolved in warm water and 30 parts sodium acetate added. It melts at 170° C.—*Pharm. Ztg.*, 1891, 628.

New alkaloid in the areca nut.—In a recent communication, E Jahns announces the discovery of a new alkaloid which he calls *guvacine* (from Guvaca, the old Indian name for the areca palm); it has the formula $C_6H_9NO_2$ and differs from *arecaine* and *arecaidine* by minus CH_2 and from *arecoline* by minus $(CH_2)_2$. It crystallizes in small, lustrous scales, easily soluble in water and dilute alcohol, insoluble in strong alcohol, ether, chloroform and benzol; at 265° C. it becomes dark in color and melts at 271–272° with decomposition. It forms salts of acid reaction having the same solubilities as the base.—(*Ber. d. D. Chem. Ges.*) *Pharm. Ztg.*, 1891, 671.

Phenacetine can be distinguished from acetanilide and antipyrine by the action of hot, dilute nitric acid. If the finely powdered phenacetine be covered with a 10–12 per cent. nitric acid and heated to the boiling point for a short time, the liquid as well as the powder

will assume an intense yellow color due to the formation of a nitrophenacetine; acetanilide and antipyrine under the same treatment are not altered.—W. Autenrieth and O. Hinsberg, *Arch. der Pharm.*, 1891, 456.

Detection of oil of geranium in oil of rose.—1. If two or three drops of oil of geranium be agitated in the cold with two cubic centimeters of a fuchsin solution decolorized by addition of sulphurous acid there will be obtained at first a bluish violet, after two hours a beautiful blue coloration. Oil of rose by the same test after 24 hours' standing shows a red coloration. With mixtures of the two oils there is no difficulty in recognizing the blue color due to oil of geranium since this reaction is produced before the color reaction due to the oil of rose.

2. If equal quantities of oil of geranium and concentrated sulphuric acid be mixed in a watch-crystal heat is developed with formation of dense, white fumes of a disagreeable tarry odor; a brown-red viscid liquid remains which on addition of 95 per cent. alcohol becomes *turbid* and separates yellow, fatty flakes, the solution at first being red but passing into a yellow after standing. With oil of rose the test also forms a brown-red viscid mass which, however, dissolves *completely* in alcohol forming an almost colorless solution. —G. Panajotow (*Ber. d. D. Chem. Ges.*) *Pharm. Ztg.*, 1891, 685.

Alkaloids of the Solanaceæ.—A careful investigation of the important plants of this natural order, with the view of ascertaining which of the alkaloids existed pre-formed in them, is summarized as follows:

Belladonna root: Young, uncultivated roots contain only hyoscyamine; older, uncultivated roots contain atropine in minute quantity with the hyoscyamine; the same alkaloids were found in older cultivated roots. *Belladonna berries:* The ripe, cultivated Atropa Belladonna nigra contain both alkaloids, the uncultivated only atropine; the unripe, uncultivated berries contain chiefly hyoscyamine, with very little atropine; the ripe berries of Atropa Belladonna lutea contain atropine with another alkaloid probably identical with atropamine. *Belladonna leaves:* Both species contain both alkaloids, hyoscyamine and atropine, the latter in minute quantity only. *Stramonium seed:* Fresh and old seeds contain chiefly hyoscyamine, with small quantities of atropine and skopolamine. *Solanum tuberosum* contains a mydriatic alkaloid along with *betaine*. *Lycium*

barbarum and *Solanum nigrum* contain mydriatic alkaloids in very minute quantity, which appear to be identical with the alkaloids of Solanum tuberosum. *Nicotiana Tabacum:* The leaves contain traces of mydriatic alkaloids. *Anisodus luridus:* The seeds, herb and root collected in autumn contain pre-formed only hyoscyamine. —W. Schütte, *Arch. der Pharm.*, 1891, 492.

Andromedotoxin.—In connection with his continued researches on this poisonous principle of certain ericaceæ, Prof. Plugge gives in *Arch. d. Pharm.*, 1891, 552, a complete list of the plants examined in this respect. The lists published in Am. Jour. Pharm., 1889, 360, 361, may now be extended as follows:

The poison is present in Kalmia angustifolia, *Lin.*, Monotropa uniflora, *L.*, Pieris formosa, *Don*, P. ovalifolia, *Don*, Rhododendron Falkoneri, *Hook.*, R. grande, *Wight*, R. barbatum, *Wallich*, R. fulgens, *Hook.*, R. cinnabar, *Roxb.*, and R. punicum, *Smith.* The poison is absent from Arbutus Andrachne, *L.*, A. canariensis, *Lam.*, A. integrifolia, Lam., A. Unedo, *L.*, Arctostaphylos alpina, *Spr.*, A. glauca *Lindl.*, Erica arborea, *L.*, Pyrola maculata, *L.*, P. rotundifolia, *Lin.*, Ledum latifolium, *Lam.*, and Rhodo. ferrugineum, *L.*

Test for cerium salts.—To the solution or salt to be tested add a slight excess of sodium hydrate and evaporate to dryness and then a few drops of a solution of strychnine in concentrated sulphuric acid (1 : 1,000); in the presence of 0·01 mg. cerium salt a faint and vanishing blue violet coloration will result; if 0·1 mg. be present at first a blue and later a permanent red coloration results. This test is the reversed strychnine reaction proposed by Sonnenschein.—Prof. P. C. Plugge, *Arch. der Pharm.*, 1891, 558.

The alkaloid of Sophora tomentosa, L., (Am. Journ. Pharm., 1891, 231) according to a preliminary note by Prof. Plugge is very likely to be proven identical with *cytisine;* the chemical as well as physiological tests with a small quantity of material gave all tests for cytisine.—*Arch. der Pharm.*, 1891, 561.

Detection of Traces of Copper in Distilled Water. By H. Thoms. —Copper to the extent of 1 : 200,000 in distilled water gave no indications with either ammonia or potassium ferrocyanide, but with potassium iodide solution a faint yellow color appeared which gave a distinct blue tint with starch paste. A comparative test with pure distilled water showed no coloration with iodide. —*Phar. Centralh.; Jour. Chem. Soc.*, May, 1891.

TUBERCULIN.

Considerable interest has been recently excited by the publication of a further communication upon tuberculin, in which Dr. Koch makes known the results of his attempts to obtain the *active principle* in a purer form (*Deut. med. Wochensch.*, October 22, p. 1189). In Dr. Koch's first attempt to purify tuberculin it was mixed with five times its volume of absolute alcohol, which caused a separation of a brown resinous mass that adhered to the bottom of the vessel. Both this deposit, however, and the clear supernatant liquid showed the tuberculin action to nearly the same degree. When a greater excess of alcohol was used instead of the resinous mass, a fine granular precipitate was thrown down, which after being washed repeatedly with absolute alcohol and dried in a vacuum over sulphuric acid, gave an almost white powder. But in this powder the tuberculin was still contaminated with extractive matter insoluble in alcohol, and many fruitless attempts were made to remove it. Either, as when ammonium sulphate was used, the tuberculin was thrown down still contaminated with other matters, or it lost its activity, or it could not be separated in an active condition from its precipitant. For instance, tannin threw down all the active principle from tuberculin, and the precipitate brought into solution by the addition of sodium carbonate possessed still its full activity, but the active substance could not be again separated from the tannin. Eventually it was found that by using a much smaller proportion of absolute alcohol (two parts to three of tuberculin) no separation of brown resinous substance was caused, but that at the end of twenty-four hours a white flocculent precipitate was formed. The brown supernatant liquid was decanted and replaced by the same quantity of 60 per cent. alcohol, the whole stirred and allowed to stand, and this was repeated until the alcohol remained uncolored, when the precipitate was washed once with absolute alcohol and dried in a vacuum exsiccator. The product was a snow-white mass, which after drying at 100° C. appeared when powdered of a light gray color, and was considered to represent the pure or nearly pure active principle of tuberculin. It dissolved fairly easily in water, but an aqueous solution appeared to lose some of its activity in a fortnight. The evaporation of such a solution also was found liable, as the concentration increased, to cause the separation of curdy flocks, which did not redissolve when more water was added. On the

other hand, a 50 per cent. solution of the purified tuberculin in glycerin appeared to be very stable. From a consideration of its chemical reactions, and the results of three elementary analyses, it would appear that tuberculin is closely allied to the albumoses, but differs from them, and especially from toxalbumen, by its resistance to high temperatures; it also differs from the peptones in several respects, especially in being completely precipitated from a solution by ferric acetate. From the results of a number of physiological experiments, made first upon guinea pigs and afterwards on human patients, Dr. Koch has arrived at the conclusion that the purified tuberculin does not differ markedly in the nature of its action from the crude tuberculin, but that the former has, in fact, diagnostically and therapeutically the same effect as the latter when it is used in such quantities that the reaction symptoms, especially that of temperature, attain the same degree of intensity. In respect to this point, it has been found that whilst the action of purified tuberculin upon guinea pigs is about fifty times as strong as that of the unpurified, upon human patients its action is at most forty times as strong. From these results, therefore, it would appear that Dr. Koch and his coadjutors have not been so successful in the attempt to purify tuberculin as Dr. William Hunter, who claims to have succeeded in removing from it the constituents giving rise to fever and inflammation, leaving a product which in the hands of Dr. Watson Cheyne has proved capable of inducing local reaction, followed by healing change around tuberculous lesions, unaccompanied by any constitutional disturbance.

In an appendix to this communication, Dr. Koch gives some further information as to the method adopted in *preparing tuberculin.* It is generally understood that tuberculin is a product of the cultivation of tubercle bacilli obtained in the form of a glycerin extract. As may be supposed, a principal difficulty has been the cultivation of the bacilli free from admixture with other organisms. Originally the tubercle bacilli were sown upon "glycerin-peptone-agar," and when the culture had attained its full development it was washed off, collected on a fine wire gauze, extracted with a 4 per cent. glycerin solution, the solution evaporated to one-tenth, filtered, and the filtrate used. When a large demand arose for tuberculin, the agar cultivation was found unsuitable, as it gave relatively small results. A previously abandoned attempt to cultivate the bacilli in a liquid

medium was therefore resumed, but at first without success, until some small flat pieces of cultivation which had been left dry and unmoistened in the upper part of the flask, were observed swimming on the top of the liquid, where they developed most luxuriantly. In the course of a few weeks they formed over the entire surface a tolerably thick whitish skin, dry on the upper side, which eventually became moistened, broke up and sank to the bottom. The product from such a culture proved considerably greater than that developed on solid media. The cultivation liquid used is an infusion of veal made faintly alkaline and with one per cent. of peptone and 4 or 5 per cent. of glycerin added. The bacilli sown give practically the same results, whether taken from fresh or old cultivations, from a tuberculous patient, or after passing through a series of animals. When the culture is quite mature, which occurs at the end of six or eight weeks, and has been ascertained to be absolutely pure, it is extracted by means of the cultivation liquid itself, and the extract is evaporated on a water bath to one-tenth of its original volume and filtered through porcelain. It then contains from 40 to 50 per cent. of glycerin, and is tested as to its activity by experiments upon guinea pigs.—*Phar. Jour. and Trans.*, Oct. 31, p. 345.

DETECTION OF FORMLESS FERMENTS AND POISONS IN BLOOD.

By Prof. R. Kobert.

Both the physiological and the chemico-legal demonstrations of unstable poisons in blood encounter difficulties depending mainly on the fact that the coloring-matter of the blood (if the red globules are dissolved by putrefaction, morbid processes, or improper treatment such as the addition of water) forms a tarry mass which it is difficult to treat chemically and which is perfectly useless for physiological experiments. The removal of this tarry coloring-matter is generally effected by plentiful dilution and boiling with acetic acid, or precipitation with alcohol, with potassium ferrocyanide and acetic acid, or with uranium nitrate.

In all these cases the serum albumen present in the blood along with the blood-pigment as well as all albuminoid enzymes and toxalbumins are simultaneously precipitated, and thus the detection and the isolation of the enzymes and toxalbumins is rendered impossible. But if we had an agent which would throw down the

coloring-matter of the blood, leaving all other substances in solution, and chemically unaltered, we should have made an important step towards the isolation and detection of the enzymes and tox-albumins.

Zinc-powder may be regarded as such a precipitant for hæmoglobin. In forensic chemistry which is generally concerned with stale, offensive blood, this agent has the advantage that it renders the specimen almost inodorous even if several weeks old. The completeness of the precipitation is not interfered with by the age of the blood. The following conditions are essential for successful precipitation :

(1) In quite recent blood, freshly drawn from normal men or other animals, which is always alkaline, the alkalinity must be neutralized before the addition of the zinc-powder. The same holds good of the blood of dead bodies a week old in which ammonia has been formed. On exposure to the air, or remaining in the body, the normal alkalinity of the blood is lost within 1 to 2 days. Diseases—especially fevers—often reduce the alkalinity before death to such a degree that the zinc may be added directly.

(2) The blood must be free from methæmoglobin. If this substance is present the blood is allowed to remain in an open vessel without dilution or shaking until the last trace of methæmoglobin has disappeared. It is known that this disappearance often ensues within 24 hours, whether in the corpse or in jars, even when the original quantity of methæmoglobin was very considerable.

(3) The blood must be diluted with at least 3 to 5 volumes of water.

(4) The zinc-powder must be as pure as possible, containing nothing but zinc and zinc oxide.

(5) The quantity of the zinc-powder must be equal to a quarter or a half of the original weight of the blood.

(6) The mixture must be energetically shaken for a considerable time.

If these conditions are observed a complete separation of the coloring-matter of the blood is almost always effected, so that even on washing the precipitate with much water in the filter-press or in the filter-pump nothing returns into solution. The yellowish-brown coloring-matter contained in the serum of some kinds of blood is not thrown down, but remains in the filtrate. Distinctly recog-

nizable quantities of zinc are to be found in the filtrate only if compounds of organic acids were present in the blood. Otherwise mere traces of zinc exist as zinc albuminate, in smaller proportions the more the zinc-powder is free from zinc oxide. As the deposit —a solid, reddish-brown, bulky mass—retains no organic poisons except traces of hydrocyanic acid and carbon monoxide, we have in the filtrate all the glycosides, alkaloids, ptomaines, toxalbumins, enzymes, amides, etc.; in short, the examination of the filtrate is exclusively important for forensic chemistry. Concerning the solid residue we need merely remark that the hydrocyanic acid may be removed from it by extraction with alcohol and carbon monoxide by exhaustion in the air-pump.

The filtrate is incomparably more suitable than the original blood as well for the chemical as for the physiological detection of poisons. If its action upon animals is to be examined it is only requisite to shake up a portion with a drop of solution of sodium sulphide, and to filter off the precipitate of zinc sulphide. If the proportions are duly adjusted the liquid, free from zinc and sodium sulphide, can be injected subcutaneously into a mouse or into the circulatory system of a kitten. However, the filtrate can often be applied without the elimination of the zinc. The bacteria which swarm in putrid blood are almost entirely left behind in the residue. Where an absolute determination of bacteria is required the filtrate may be again passed through a Chamberland filter, and thus the last residues of the germs may be removed. If the animal experimented upon exhibits grave phenomena of poisoning, a second portion of the original filtrate is mixed with a drop of solution of potassium ferrocyanide and a little acetic acid, the precipitate, which includes all the zinc and the albuminoids, is filtered, the filtrate is neutralized and injected into a second animal. If this animal, in contradistinction to the former, exhibits no phenomena of poisoning, the symptoms observed in the former subject must have been occasioned by an albuminoid poison, as in such dilute liquids other poisons are not precipitated by potassium ferrocyanide, or at most a portion of strychnine, which can be easily detected in the precipitate.

The isolation of the poisonous albuminoid substance can be undertaken only in the main portion of the original filtrate, either by treatment with alcohol in the ordinary manner or by "salting out" with ammonium sulphate. In those toxalbumins, however,

which do not admit of the above-mentioned methods of precipitation, *e. g.*, those of venomous spiders, a more complete isolation of the poison is not yet practicable.

If the second experimental animal does not remain in health, but is taken ill like the first, we have a proof that the poison in question does not belong to the albumins—at least not to those precipitable by ordinary means. The entire filtrate is then freed from albumin and the limpid filtrate is examined for other poisons by Dragendorff's method.—*Chemiker Zeitung; Chemical News*, Oct. 23, 1891, p. 206.

THE SEPARATION OF RESIN FROM FATTY ACIDS.

By J. Arthur Wilson.

The detection of resin and its separation from fats and oils was at one time a very difficult problem to the analyst, and in many cases was impossible. Amongst many processes we have those of Henderson, Barfoed, and Gladding, the latter of which has found the greatest favor. It suffers from one very serious defect, however, and that is the correction for solubility of the fatty acids and silver salts in ether. This is a very variable factor, and, of course, is dependent on the nature of the fatty matter used in the fabrication of the soap. With small quantities of resin the error thus introduced may be as much as 20–30 per cent., but of course in soaps of high resin content—say 20 per cent.—the error diminishes.

Mr. R. Williams (*Analyst*, vol. xv., p. 169) obtained very good results by the process; thus, in a soap which actually contained 30·0 per cent. of resin, he obtained 30·7 per cent., but no mention is made as to whether the amount remaining in the lyes, after precipitation of the soap, was determined. As to the simple detection of resin, no doubt the best test is to boil the dry fat or fatty acids (10 drops) with 5 cc. acetic anhydride, cool, and add 2 drops of pure sulphuric acid (1·84 specific gravity), when the reddish violet color due to resin is easily observed, one per cent. of which can thus be detected.

It seems that all the above processes must make way for the admirable process of Twitchell (*Analyst*, Oct., 1891), who found that whereas fatty acids are, as is well-known, converted into eutral ethers on passing dry hydrochloric acid gas into their alco-

holic solution, the acids constituting resin are not attacked. The process obviously may be either gravimetric or volumetric, but chemists will prefer the latter on account both of speed and accuracy. The description of the process is as follows:—2·5 to 3 grms. of the dried fatty and resin acids are dissolved in from 25–30 cc. of perfectly absolute alcohol, and a current of dry hydrochloric acid gas passed in constantly. The flask containing the mixture must be kept cool by immersion in cold water. When the etherification is complete the ethers separate and the gas is no longer absorbed. The flask is removed, corked well, and allowed to stand half an hour. It is diluted with about 100 to 125 cc. hot water, then cooled, transferred to a separatory funnel, and agitated with 75 cc. of ether. The watery layer is removed, and the ethereal layer washed with cold water till neutral to delicate litmus solution (twice always is sufficient). The ethereal liquid is received in a flask, the separator washed out with about 50 cc. of neutral spirit, and the whole titrated by standard alcoholic alkali, using phenolphthalein as indicator. The alkali should be standardized by an accurate solution of half normal HCl, and should be either one-fifth or one-half normal according to the amount of resin in the fatty acids. Taking the mean combining weight of resin as 347, then the percentage is easily calculated; it is reported both on 100 of dry fatty matter and on 100 of soap.

Twitchell's test experiments are eminently satisfactory, and I have now pleasure in confirming them by my own tests:

(1) Sample of distilled fatty acids containing no resin, when treated as described showed 0·7 per cent. resin.

(2) The above distilled fatty acids, to which were added common resin to the extent of 22·5 per cent., showed, when treated as above described, 23·3 per cent. of resin, which, after deducting the 0·7 per cent. found in No. 1, gives exactly 22·5 per cent., or the amount which was added.

(3) Soap made from palm oil and other fats, but no resin, gave 0·8 per cent. resin.

(4) Another soap made from fatty matter of unknown origin, but no resin, gave 1·0 per cent. resin. This, it should be observed, was of a dark color, and evidently contained some altered or oxidized oily matter.

(5) Soap which showed by Gladding's test 6 per cent. of resin, gave 4·9 per cent. by Twitchell's process.

The above results are all that may be desired, and I have now to show how the process may be still further shortened without any sacrifice of accuracy. This can be done by leaving out the washing and dissolving in alcohol direct. A few drops of methyl orange are then added and alkali till neutral to this indicator; phenolphthalein is then added, and the titration completed as before. The alkali required in the first case is for the neutralization of the free hydrochloric acid, and is of course neglected. That required to neutralize to phenolphthalein is, of course, due to the resin, and is calculated as such.—*Chemical News*, Oct. 23, p. 204.

TOTTINGTON MILLS, Oct. 6, 1881.

MINUTES OF THE PHARMACEUTICAL MEETING.

PHILADELPHIA, November 17, 1891.

The meeting was called to order, and Dr. C. B. Lowe was elected Chairman. The minutes of last meeting were read and approved.

Mr. Beringer presented a specimen of the flower of the vanilla plant for the museum.

The registrar presented a number of works from Prof. Maisch, and the report of the Geological Survey of the State of Pennsylvania, in four volumes, from Prof. Sadtler.

G. M. Beringer, Ph.G., read a paper upon *Synthetical Carbolic Acid*, by Dr. H. W. Jayne, who was unable to be present.

Mr. Witzel, of Tacony, exhibited a *tablet machine* and demonstrated its working facilities before the meeting.

Joseph W. England, Ph. G., read a paper on *Fluid Extract of Triticum repens*, or couch grass. It was remarked that couch grass had been greatly lauded in medical journals some eight or ten years ago, and then lost sight of, and was now being revived; there is still a large demand for it as one person has an order for three tons of it. Prof. Remington said that the specific gravity was not a very good test of commercial fluid extracts, as each manufacturer was a law to himself as far as the menstruum was concerned, and that there is a large consumption of the drug.

Mr. Thompson said agricultural journals stated that sheep seek it out in pastures, and that they are relieved in passing biliary calculi. Prof. Maisch stated that in Europe there are two preparations in use, a soft extract and a liquid extract made by dissolving the former in a little water or weak spirituous liquid; the drug had been popularly employed for many centuries and was generally given in decoction.

J. W. England, Ph.G., spoke of *Ehrlich's Test for Typhoid Fever Urine.* It consists of two solutions: Solution A is made by dissolving one part of *nitrite of sodium* in one hundred of distilled water; Solution B, by mixing 10 cc. of chemically pure hydrochloric acid with 190 cc. of distilled water, and adding sufficient sulphanilinic acid to saturate, about two and a half grammes will be requisite. It is used by adding a small quantity of Solution A to the suspected

urine, and then Solution B following with ammonium hydrate ; should a typhoid condition exist, it will occasion a wine coloration.

G. M. Beringer, Ph.G., read a paper upon *Mullein Oil* and its uses.

F. X. Moerk, Ph.G., read a paper upon the *Colorimetric estimation of Vanillin*, showing the great delicacy of this test. The question was asked whether vanillin from artificial sources could be distinguished from that obtained from the fruit. Mr. Moerk said it could not. Fluid Extracts of Vanilla which had no foreign color added to them gave the color reactions.

A letter from A. L. Beck, Ph.G., inquiring what should be the strength of *dilute Lactic Acid*, was read. The U. S. Pharmacopœia only gives the 75 per cent. as officinal ; two different makes which he had examined gave respectively 36 per cent. and 6·3 per cent. of absolute acid. He says that he has made it 10 per cent. to have it uniform with the other dilute officinal acids ; he expresses the opinion that manufacturers should indicate the strength of the preparation on the label.

J. W. England read a note upon *salicylate of bismuth.* A motion was made to refer the papers read to the Publication Committee. Mr. England called attention to the use of *Menthol and Carbonate of Ammonia* in colds in the head, hay fever, etc., to be employed as smelling salts ; they should be mixed in about equal quantities. Dr. Miller said that it was important that the carbonate of ammonia should not be in too great excess.

Inquiry was made why *abstract of belladonna* was made from the root when the pharmacopœia directed the extract and tincture to be prepared from the leaves ; the reason was that in preparations so well-known as the extract and tincture, any change would produce great dissatisfaction, while it is well-known that the root is far more active and hence was employed.

On motion, adjourned.

T. S. WIEGAND,
Registrar.

REVIEWS AND BIBLIOGRAPHICAL NOTICES.

The P. C. P. Alumni Report.—Published under the auspices of the Alumni Association of the Philadelphia College of Pharmacy.

Heretofore the Report of the Alumni Association made its appearance once a year. With the present season a change has been made, so that a report will reach the alumni eight times a year, each containing the minutes of the last social meeting, and information on other College matters. The publication is in charge of Jos. W. England, Ph.G., as editor, and Wm. Nelson Stem, Ph.G., as business manager. The first issue contains 24 pages, and including a number of editorial notes and discussions, makes attractive and interesting reading matter. The periodical is published at the subscription price of $1 a year.

Exercise Book of Junior Pharmaceutical Laboratory Work.—Division one. Arranged for use in the Massachusetts College of Pharmacy, Boston, Mass. By Edgar L. Patch, Ph.G., Professor of Theory and Practice of Pharmacy, and Director of Pharmaceutical Laboratory. 1891.

A very conveniently and systematically arranged book, containing typical processes and manipulations arranged for the practical instruction of tyros in pharmacy. Beginning with the testing of thermometers and the taking of

specific gravities under various conditions, the exercises proceed to the exsiccation of salts, the purification of crystals by recrystallization, and the preparation of chemical solutions, crystals, tinctures, fluid extracts, syrups, plasters, etc., and conclude with the reading and dispensing of prescriptions. As will be observed it is a work for practical use, and it is designed to be used as a note and memorandum book, being interleaved, and upon the printed pages, at the end of each exercise, provided with a series of printed memoranda, containing blanks to be filled out by the student. Aside from the usefulness of these memoranda as a check upon the correct execution of the work, they will inculcate habits of observation, which must be of lasting value to the students in the future.

Twenty-second annual Report of the State Board of Health of Massachusetts. Boston: 1891. 8vo. Pp. 588.

The publication contains reports on water supply, sewerage, food and drug inspection, mortality, etc. Reports on food were made by Dr. Harrington and Davenport; on milk by Prof. Goessmann, Davenport and Worcester; and on drugs by Prof. Davenport.

History of Circumcision from the earliest times to the present; moral and physical reasons for its performance, with a history of Eunuchism, Hermaphrodism, etc., and of the different operations practised upon the prepuce. By P. C. Remondino, M.D. (Jefferson), etc. Philadelphia and London: F. A. Davis. 1891. 12mo. Pp. x and 346. Price, cloth, $1.25 net.

The title, as given above, explains the scope of the work, which deals with a custom of great antiquity, having originated under certain climatic conditions, and being practised to some extent as a religious rite, but much more extensively as an inherited custom and, according to the author, in modern times also as a measure of hygiene. The subject is treated with delicacy and frankness, the historical information being of general interest, also the hygienic discussions, while obviously the chapters relating to surgical operations and the like are intended especially for physicians. The book forms No. 11 of the Physicians' and Students' Ready Reference Series.

Ueber Einwirkungen des Cyanwasserstoffs, des Chloralhydrats und des Chloralcyanhydrins auf Enzyme, auf keimfähige Pflanzensamen und auf niedere Pilze, von Ed. Schär, Professor der Pharmacie am Eidgenössischen Polytechnikum in Zürich. Zürich: Albert Müller's Verlag. 1891. Fol., p. 25.

On the effects of hydrogen cyanide, of chloral hydrate and of chloral cyanhydrin upon enzymes, upon fertile plant-seeds and upon some low fungi.

An interesting series of experiments demonstrating that certain seeds are not prevented from germinating when under the influence of solutions of definite strength of the chemicals named, while other seeds showed little or no power of resistance in this respect. The essay forms one of the contributions to the jubilee volume, issued by the higher educational institutions of Zurich, in commemoration of the fiftieth anniversary of the doctorates of Professor Carl Wilhelm von Nägeli, in Munich, and of Professor Albert von Kölliker, in Würzburg.

All around the Year.—A calendar for 1892. Designs by J. Pauline Sunter. Boston: Lee & Shepard. Price, in box, 50 cents.

This calendar is composed of heavy, gilt-edged cards, 4¼ x 5½ inches,

tastily tied with white silk cord, and a delicate silvered chain attached, by which they may be hung on the wall or elsewhere, and are so arranged on rings that they may be turned over as each month shall be needed for reference. The designs are appropriate and quite attractive, and in each case suggestive of the season represented.

The Physicians' Visiting List for 1892. Philadelphia: P. Blakiston, Son & Co. Price, for 25 patients, $1.

This is the forty-first year of the issue of this Visiting List, and it is arranged in the usual convenient style. The several tables contained in the preliminary part, are each credited to some one, with the exception of a "Table for converting apothecaries' weights and measures into grams," and this happens to be an old acquaintance; for fifteen years ago we made the calculations and published the tables in the Amer. Jour. Phar., 1877, p. 92, and they have also since been published in the National Dispensatory, and in many other publications—not unfrequently without proper credit having been given. This has not, however, been detrimental to their usefulness.

Hand-book of Materia Medica, Pharmacy and Therapeutics, including the physiological action of drugs, the special therapeutics of disease, official and practical pharmacy, and minute directions for prescription writing. By S. O. L. Potter, A.M., M.D., etc. Third edition, revised. Philadelphia: P. Blakiston, Son & Co., 1891. 8vo. Pp. 767. Price, cloth, $4; full leather, $5.

In the present edition some new remedies, like creolin and somnal, have been added in the part treating of materia medica, several changes have been made in the arrangement, and some of the articles have been partly rewritten. On comparing this with the previous edition, we must repeat what we then said in this Journal, 1890: "Most of the errors which we noticed in the first edition, to some of which we referred in the previous review, remain uncorrected. * * * The general correctness of all that belongs to the therapeutical uses and the physiological action of medicines, will render the book of as great practical value to the physician, as the preceding edition has been."

Copies of the printed Proceedings of the following State Pharmaceutical Associations have been received:

Louisiana.—Ninth annual meeting, held at New Orleans, April 29 to May 1. Pp. 138. See July number, p. 370. The next meeting will take place in New Orleans, April 13, 1892. Mrs. E. Rudolf, Corresponding Secretary.

Virginia.—Tenth annual meeting, held at Roanoke, September 8 and 9. Pp. 58. The officers for the current year are: M. E. Church, Falls Church, President; C. B. Fleet, Lynchburg, Secretary, and C. H. Lumsden, Lynchburg, Treasurer. The next meeting will be held at Fredericksburg, September 13, 1892. M. C. Hall, Local Secretary.

Wisconsin.—Twelfth annual meeting, held at Milwaukee August 11 to 13. Pp. 75. The pamphlet contains, in addition, the tenth annual report of the State Board of Pharmacy, occupying 48 pages. The officers for the present year are: C. Widule, Milwaukee, President; W. P. Clarke, Milton, Treasurer, E. B. Heimstreet, Janesville, Permanent Secretary. The next meeting will be held at Oshkosh, August 9, 1892.

OBITUARY.

William G. Buchanan, Ph.G., died in Germantown, Philadelphia, October 28; he graduated in 1862, and was for many years in the employ of Rosengarten & Sons.

George D. Coggeshall, Ph.G., died at Orange, N. J., November 5, aged 84 years. For many years he had been the oldest living Graduate in Pharmacy in the United States, having graduated at Philadelphia in 1828. He was active in organizing the New York College of Pharmacy, served that institution faithfully for many years in various positions, and was one of its representatives at the Convention of Colleges of Pharmacy in 1851, and also at the National Pharmaceutical Convention in 1852, serving in that year as the first recording secretary, and in the year following, 1853, as first vice-president of the American Pharmaceutical Association. For many years preceding his death he had retired from active business.

Edward Donelly, Ph.G., M.D., died at Piedmont, Cal., in November, in the seventieth year of his age. He was born in Londonderry, Ireland, and came to Philadelphia in 1831, accompanying his widowed mother. He graduated in pharmacy in 1843, and afterwards joined the exploring expedition of Passed Midshipman I. G. Strain, U.S.N., returning to the United States in 1852. He graduated in medicine in 1854, and during the civil war served as surgeon with high merit. After the war he went to Pittsburgh, and in 1879, he removed to San Francisco, where he continued in practice as a physician until a few months before his death. Volumes XV and XXIII of this Journal, contain two papers—on poke root, and on cowrie resin—from Dr. Donelly's pen, and the Proceedings of the Amer. Phar. Assoc., 1860, contain a valuable paper on Theobroma Cacao, which was intended as the precursor of some other papers, that remained unfinished at the breaking out of the war.

Howard Grant Jones, Ph.G., died near Oxford, Pa., November 20, aged 39 years. Born and educated in Philadelphia, he learned the drug business with his father, Daniel S. Jones, and graduated with high honors in 1875. Afterwards he devoted his time chiefly to the study of chemistry, mineralogy and geology, the studies, however, being interrupted by a severe attack of brain fever. With returning health he resumed his mineralogical excursions, and it was upon one of these that he was suddenly prostrated.

Professor Dr. Eduard Reichardt died at Jena, October 26, aged 64 years. He was born at Camburg, became a pharmacist, studied at Jena, where, in 1854, he accepted a professorship at the Pharmaceutical and Agricultural Institute, became connected with the University of Jena as private lecturer in 1854, and in 1862 was called to the chair of technological and pharmaceutical chemistry. In 1855 he had published a valuable monograph on the chemical constituents of the cinchona barks, and in 1860 one on the Stassfurt mines. Later on his investigations were devoted to agricultural chemistry, to disinfectants, to potable waters, etc. After the death of Professor Hermann Ludwig in 1873, Professor Reichardt was elected his successor as editor of *Archiv der Pharmacie*, and continued as such until the close of 1889, when he retired from these editorial labors.

Professor Dr. Victor von Richter died suddenly in Breslau, October 8, in the fifty-first year of his age. He was the son of a Lutheran pastor in Doblen, Courland, Russia; studied physics and chemistry at Dorpat, was assistant to Mendelejeff from 1862 to 1872, and for two years professor at the Academy at Nowo-Alexandria, Poland; in 1875 he became private lecturer, and since 1879, professor of chemistry, at the University of Breslau. The deceased was an indefatigable investigator in synthetical chemistry; but his name is most familiar as the author of a work on inorganic chemistry, and one on the chemistry of the carbon compounds, both of which have been translated into several languages; one of these was only recently noticed in our October issue, page 517.

VARIETIES.

Bleaching of Beeswax; Composition of White Wax. By A. BUISINE and P. BUISINE.—In the bleaching of beeswax, light is the chief factor, since the bleaching goes on in a vacuum, or in an atmosphere of carbonic anhydride, nitrogen, etc., but ceases in the dark even in an atmosphere of ozone. Pure beeswax becomes brittle when bleached by exposure; and to prevent this, it is customary to add 3–5 per cent. of suet, which also expedites the bleaching process; the addition of turpentine oil has a similar effect. Other agents made use of are: Potassium permanganate, potassium dichromate, hydrogen peroxide and animal charcoal.—*Bull. Soc. Chim.* [3], **4**, 465–470.

Estimation of Boric Acid in Milk and Cream. By C. E. CASSAL.—The usual tests suffice for the detection of boric acid and borates in milk and cream, but for their estimation the milk or cream is rendered alkaline, evaporated and incinerated, and successive small quantities of methyl alcohol are distilled from the ash previously acidified with acetic acid. The distillate is collected on a known quantity of ignited lime, which is subsequently re-ignited and reweighed: the increase in weight is boric acid.—*Analyst*, **15**, 230–232.

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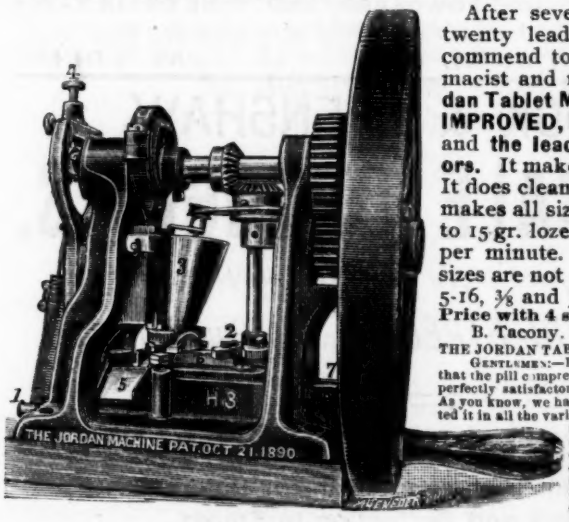

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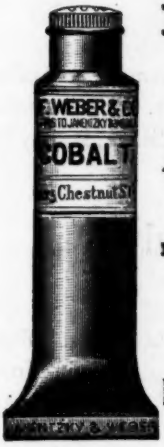

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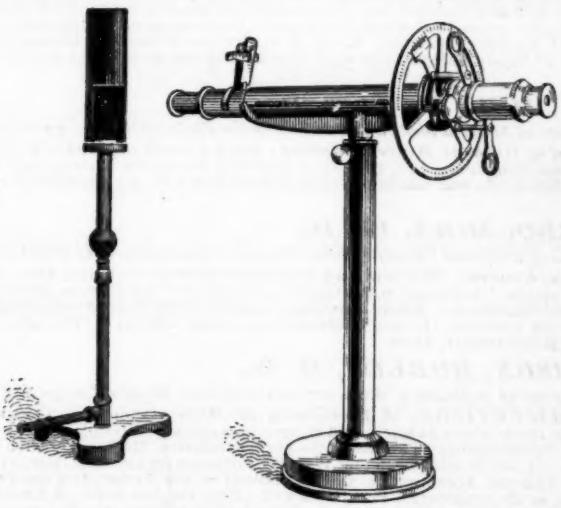

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